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THERMOMECHANICAL PROCESSING B TITANIUM

Technical Report by

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FOREWORD

This report covers the work done in the period May 11, 1972 to August 10, 1973 under the general title "Thermomechanical Processing ß Titanium". The work was sponsored by the Army Materials and Mechanics Research Center, on Contract No. DAAG46-72-C-0165, D/A Project 1W564603D385, AMCMS Code 554C.12.62000. Mr. A. Zarkades acted as Contracting Officer's Representative. The research described was conducted at Brown University by D. H. Avery and N. W. Polan.

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ABSTRACT

The results of this investigation include a set of criteria for the design of very strong yet ductile materials. By application of these criteria, thermomechanical processing has resulted in the attainment in a β -titanium alloy of highly reproducible tensile strengths in excess of 320,000 psi with a cup-cone ductile shear failure of 15% RA, exhibiting ductile behavior at elastic strains greater than 2%.

The response of the β alloy TS6 (Ti: 10 Cr, 7 V, 3.5 Mo, 3 Al), to a wide variety of thermomechanical treatments has been investigated, including the effects of cold work on polygonized structures, the effects of cold-reduction temperature, and multi-stage aging. Combinations of strength and ductility can be manipulated within a wide range by varying the amount of cold work and the aging treatment. Mechanical properties are strongly dependent upon the mode of deformation during cold working and slight variations in alloy chemistry. Stress corrosion tests in H₂0-3%NaCl and CH₃0H-1%HCl indicate superior performance of TS6 compared with similarly treated β-120VCA.

A scale-up of ingot melting and processing techniques has been accomplished. Ingots of TS6 weighing twenty-eight pounds have been prepared using a masteralloy technique. Hot extrusion at 1800°F has yielded two-inch diameter rod which has been successfully cold press-swaged 20% and cold rolled 90%. Compared with previous one-pound heats of TS6, this material overages earlier, at somewhat lower hardness levels, and with more rapid kinetics.

INTRODUCTION

The fabrication of non-equilibrium microstructures is a fresh approach to materials design which has been relatively neglected but which can produce substantially improved physical properties. The development of materials for the maximization of one property or the optimization of a combination of properties is based on the controlled placement of the constituents of the microstructure. involving new processing techniques to overcome the restraints imposed by the phase diagram and traditional casting and heat treating methods. One aim in the design of non-equilibrium microstructures is to optimize and orient the desired properties in the final fabricated item. Critical values of specific properties including strength, elastic modulus, fracture toughness, electrical conductivity, and magnetic permeability are generally needed only in specific directions in an object. This approach has been used in utilizing crystallographic texture and directionality, as in the maximizing of the permeability of silicon-iron transformer sheets; similarly, the elastic modulus in the plane of a panel subject to buckling could be maximized. Other familiar exploitations of non-equilibrium microstructures include the ausforming and patenting processes in steels.

The research described below is an example of the results of such an approach—the design of a very strong, highly ductile titanium alloy through the development of a very fine cellular dislocation structure which is amenable to low temperature aging treatments. The investigation of thermomechanical processing of the β-titanium alloy TS6 (Ti: 10 Cr, 7 V, 3.5 Mo, 3 Al) continued under this contract has resulted in further characterization, manipulation, and improvement of the properties of the material. Highly reproducible tensile strengths in excess of 320,000 psi with cup—cone ductile shear failures of 15% RA have been attained. The response to a variety of thermomechanical treatments has been studied, concurrent with the development of a set of criteria which

apply to this alloy and characterize in general the optimum design of very strong, highly ductile metallic alloys through the development of ultra-fine grain size. A scale-up of melting and processing techniques was initiated which will result in larger-scale section sizes consistent with structural applications. A preliminary investigation of the effects of slight variations in alloy chemistry has been completed, and stress corrosion properties of the alloy have been characterized.

SCALE-UP PROGRAM

The β-Ti alloy that we are developing has exhibited such superior mechanical properties that an effort has been made to scale up current melting and processing techniques to yield larger-scale section sizes consistent with structural applications. In the past we have been limited to small tensile specimen sizes—up to .160 inch diameter—since the as-received ingots have been in the form of one-pound hot-forged rods less that 1/2 inch in diameter and the subsequent mechanical deformation has been large. The scale-up program being pursued promises to yield 3/8-inch diameter rod after a 95% reduction of area through cold swaging; this material will have sufficient section size to investigate the effects of thermomechanical processing on notch toughness and to check the correspondence of scaled-up tensile properties.

Two twenty-eight pound ingots of TS6 have been prepared at AMMRC by consumable-electrode melting. The first was received during August, 1972, and was found by X-ray radiography to contain the molybdenum constituent of the alloy distributed along the axis of the casting in the form of cylindrical pellets which remained unmelted during ingot preparation. The second ingot was prepared by first combining the 10% chromium and 3.5% molybdenum constituents of TS6 to form a master alloy with a melting point of 1860°C rather than the 2625°C

and 1900°C of Mo and Cr separately. This alloy is brittle and can be reduced to chips or powder which is then readily dissolved in the remaining constituents of TS6. This method proved successful, and the second twenty-eight pound ingot was received in December, 1972. A one-inch thick disc was cut from the bottom of the ingot and sectioned into one-inch cubes. A forging study was conducted on these using a hand-operated hydraulic press at temperatures between 1950° and 1300°F. At 1800°F the yield strength was observed to be less than 15 ksi but rose above 75 ksi as the testing temperature was lowered below 1500°F. All specimens were reduced about 50% and showed no signs of crack formation at the low rates of loading employed; however, the large increase in yield strength suggests that α precipitation must be avoided by forging at 1600°F or above.

The second ingot was then machined to 4.750 inch diameter, vacuum sealed in a steel can and successfully extruded to two-inch diameter at 1800°F by Nuclear Metals, Inc. of Concord, Mass. A two-inch length was cut from the trailing end of the extrusion. The can was removed with a solution of HN₃-H₂0, and two specimens were machined and rolled without difficulty to 90% reduction of thickness. One was first solution treated at 850°C. Then both were aged at 425°C with hardness measurements taken at intervals as shown in Figure 1. Compared with heats #1 and #2 (one-pound heats), heat #5 (this twenty-eight pound heat) appears to overage earlier, at somewhat lower hardness levels, and with slightly more rapid kinetics. Otherwise, the scale-up has been accomplished quite satisfactorily, and high workability has been retained.

The entire rod was then solution treated at 850°C for one hour, water quenched, pickled to remove the steel can, and lightly surface ground to two-inch diameter. The final stage of cold working to 3/8-inch diameter rod is now being pursued but has not yet been accomplished. Two major problems encountered

have been the difficulties in scaling up ingot preparation and the unavailability of large-scale cold working facilities or the reluctance of private industries to attempt heavy cold reduction of titanium alloys using their equipment. The first problem has been solved, but it delayed further development until mid-December 1972 when the first satisfactory large ingot was produced.

The second problem was anticipated before the proposal of this project, and many industries with the capability of large scale cold working--either rod rolling, swaging or drawing--were contacted. Several of these expressed interest in undertaking the work, believing it to be within their capacity. Now that the material has been prepared, however, and formal commitment to assume the project is sought, several new difficulties have arisen. Previous experience with titanium alloys in general has prejudiced many possible sources against attempting the work despite our own extraordinary results with cold reduction of both the one and twenty-eight pound ingots of TS6. Most of those who do feel that they may have the capacity and are willing to make the attempt require equipment breakage guarantees and patent rights from this contractor, both of which are unacceptable.

Of the major firms contacted, including U. S. Steel, Alcoa, Wyman-Gordon, Bethlehem Steel, Washburn Wire, and Carpenter Technology, only two agreed to satisfactory terms within the financial limitations of this contract. Wyman-Gordon attempted to cold forge a section of the two-inch diameter rod to one-inch diameter which would then be cold extruded to 3/8 inch using available equipment. This attempt was unsuccessful, however, since only flat forging dies were available, producing a completely unacceptable state of stress in the material resulting in fracture after less then 15% RA.

The second alternative--use of a 1000 ton press and large capacity swaging equipment at the Bethlehem Steel Co.--has also been pursued and has demonstrated

that cold reduction of the scaled-up material is, indeed, feasible. The two-inch diameter rod was reduced 20% RA by cold press-swaging, deformation of the rod being homogeneous with no cracking occuring. Further reduction was not attempted due to damage to the relatively soft R_c43 press dies which deformed plastically during loading. The available rotary swaging equipment also proved inadequate since the included angle (8°) of the dies was excessive. Nevertheless, given appropriately designed equipment, cold reduction of TS6 on a large scale seems commercially practical.

While scaled-up thermomechanical processing has been accomplished, it is our opinion that the most appropriate application of this material does not lie in large scale structures but in fabrication on a smaller scale where the required mechanical operations are quite readily and efficiently obtained—high strength wire cable and fasteners are prime examples. If large scale applications are to be pursued, in view of the experiences indicated above, separate industrial contracting to develop the capacity for heavy cold deformation of large section sizes is required.

OPTIMIZATION OF MECHANICAL PROPERTIES

The investigation of the mechanical and stress corrosion properties of the alloy was conducted on the one-pound ingots produced at AMMRC (heats #2 and #3). While the results presented below are extraordinary, representing ductile behavior in metals at elastic strains in excess of 2.3%, they are not, we believe, unique to this specific alloy. Rather they represent the behavior of a particular ultrafine fibrous microstructure previously exploited only in heavily drawn pearlite. Concurrent with the empirical investigation, we have proposed a set of criteria for the characterization of an optimal material for high strength and high ductility through the development of ultra-fine grain size. We now

outline the rationale for these criteria and discuss the results of their successful application to the particular case at hand-the thermomechanical processing of β titanium.

Design criteria

- 1. The starting material consists of a metastable fcc or bcc single phase.
- 2. The material has a low strain-hardening exponent and good workability.
- 3. In order to form stable subgrain cells the material has a low stackingfault energy and a high recovery temperature.
- 4. A hardening mechanism active below the recovery temperature is available, i.e. metastable decomposition or local ordering, not solid-solution diffusion-controlled precipitation.
- 5. The flow stress is relatively insensitive to temperature.

Rationale

The central requirement of this analysis is the development of a very fine subgrain cell structure which is kinetically homogeneous and responds to a relatively low-temperature hardening mechanism. Work hardening must be discounted at the outset since the criterion for the maintenance of mechanical stability-- $d\sigma/d\varepsilon$ > σ --becomes impossible at very high stresses. If this criterion were to be met, after very small strains it would impose stress levels in excess of any conceivable fracture stress. However, if a very fine-grained cellular substructure can be produced, dislocation motion is limited and the yield strength raised according to the Hall-Petch relation,

$$\sigma_{y} = \sigma_{y_{0}} + \frac{K_{y}}{\sqrt{d}}.$$

Simultaneously, the fracture strength is increased by

$$\sigma_f = \sigma_f + \frac{K_f}{\sqrt{d}}$$

if microstructurally dimensioned microcracks or voids are the controlling fracture mechanism. Such a microstructural strengthening was first observed and exploited in heavily drawn two-phase pearlite [1,2]. Under the proper conditions, however, plastic deformation of single-phase material results in the formation of a stable cell structure which can be reduced in dimension upon further deformation. The critical dimension is then given by

$$d = d_0 \sqrt{1 - CW} ,$$

where CW is the reduction of area due to cold working (e.g. swaging or drawing) the material and d_0 is the hypothetical initial subgrain size as shown schematically in Figure 2. Then

$$\sigma_{y} = \sigma_{y_{0}} + K'_{y}(1 - CW)^{-1/4},$$

and

$$\sigma_{f} = \sigma_{f_{o}} + K_{f}(1 - CW)^{-1/4}$$

The development of stable effective substructures generally requires cold deformations of at least 30% to 40%; and, since the desired fine submicron cells require further reduction, good workability is essential. For this reason, this type of processing is considered to be limited to fcc or bcc solid solution alloys with a relatively low strain-hardening exponent.

In order to stabilize and maintain the cell structure, a low stacking-fault energy and high recovery temperature are necessary. Assuming deformation induced transformations have been avoided, the highly deformed material is essentially kinetically homogeneous and amenable to further strengthening through transformation or ordering of the metastable starting material. However, any thermal hardening mechanism must occur without altering the cell geometry. Therefore, only metastable decompositions, interstitial diffusion, and local

ordering reactions seem feasible, and solid solution diffusion-controlled precipitation mechanisms are excluded. A material which satisfies these criteria can be heavily cold worked at a moderately low yield strength to develop the substructure; subsequent aging precipitates a second phase to produce maximum strength with the retention of some ductility since no preferential segregation occurs at grain boundaries.

The desirability of a low sensitivity of flow stress to temperature is dictated by the attempt to avoid low-energy-absorption adiabatic shear fractures. For a material showing exponential hardening-- σ = $K\epsilon^n$ --in a first order approximation completely adiabatic flow would locally reduce the yield strength to

$$\sigma = K \varepsilon^{n} \left[1 + \frac{\varepsilon}{c_{p}(n+1)} \right] \frac{d\sigma}{dT} .$$

where c_p is the heat capacity. Thus we favor a low value of $\frac{d\sigma}{dT}$. Optimally, large c_p and n are desirable also. However, most high strength metals are characterized by a low strain hardening exponent; and, since we previously required good workability, some tendency toward localized non-cleavage type deformation must be anticipated.

Application of the criteria to β-titanium alloys

Promising candidates for this type of thermomechanical strengthening treatment include the metastable β alloys of titanium. In these alloys, the high temperature bcc phase is retained at room temperature by rapid quenching. Subsequent aging can permit precipitation hardening by decomposition of the metastable β phase. A common problem, though, is preferential precipitation at grain boundaries, with the attendant degradation of strength, ductility, and stress corrosion properties. Deformation prior to aging tends to produce a kinetically homogeneous material and is the basis for ordinary thermomechanical

treatments. Lerinman, et al. [3] reported work in which the alloy TS6, of composition Ti: 10.8 Cr, 7.8 V, 3.4 Mo, 3.2 Al, responded favorably to thermomechanical treatment. Cold reductions up to 40% and subsequent aging to precipitate the finely dispersed hcp α phase produced a maximum ultimate tensile strength of 234,000 psi compared with the solution treated and quenched strength of 119,000 psi. The stacking-fault energy for the alloy was determined to be very low, approximately 15 erg/cm² [4]. These were among the earliest investigations of thermomechanical processing of the β -Ti alloys, and, in view of the results, TS6 was chosen for study in this investigation. We believe, however, that the entire class of β alloys is susceptible in some degree to the treatment described here. For example, TS6 is similar to Crucible β -120VCA.

Several one-pound ingots of composition similar to TS6--Ti: 10 Cr, 7 V, 3.5 Mo, 3 Al--were produced at AMMRC by conventional titanium melting techniques--nonconsumable-electrode arc melting followed by forging and remelting in a consumable-electrode arc furnace. These ingots were then hot forged and swaged to 0.4-inch diameter rods, machined to 0.350 inch diameter, solution treated in vacuo at 850°C for 1/2 hour, water quenched, and variously cold swaged or drawn up to 96% RA with no resultant defects and no intermediate annealing treatments. The elastic modulus is low--between 14 and 16 × 10 psi, depending on preferred orientation. The strain-hardening exponent is also low, having been determined by hardness measurements to be 0.16. Transmission electron microscopy on the cold worked material [5] shows dislocation tangles growing in density and irregularity as deformation increases. Few twins are found and no transformation induced phases are seen. The most heavily worked material has an irregular cell structure of approximately 0.05 to 0.08 µ diameter.

Results from unaged specimens tested in uniaxial tension support the above analysis. In Figure 3a, yield strength plotted as a function of $(1 - CW)^{-1/4}$,

the measure of the effective cell size, agrees with the Petch hardening equation. At deformations up to 96.2% RA, the limit of the present investigation, little tendency for spontaneous recovery is exhibited. It has been observed, however, in similarly heavily cold-drawn chromium [6], that the subgrain size decreases rapidly up to 98% RA but stabilizes thereafter.

Aging the highly deformed material between 350 and 500°C produces a fine, uniformly dispersed a precipitate. Representative tensile data for a variety of thermomechanical treatments of two different heats of material are presented in Table I and Figure 4. In even the very high strength condition, failure occurs by void growth and ductile shear as illustrated in Figures 5 and 6.

The overall results from heat #3 show an increase of about 20% over previously obtainable tensile strengths and were rigorously examined to assure their validity. Testing equipment was carefully calibrated and monitored; specimens were repeatedly measured using a 0.017-inch blade micrometer and two measuring microscopes; data for the high strength aged specimens were reproduced in three separate test series. The order of machining and aging proved irrelevant—aging the as-machined surface at 425°C in air has no adverse effects on mechanical properties. While the drawn wire exhibits cup—cone fracture, the swaged wire aged to high strength shows observable necking with wolf—ear fractures. On some specimens this precluded the measurement of RA's as seen in Table I.

The enhanced properties of the swaged material from heat #3 are due to a combination of the mode of deformation and alloy chemistry. The maximum flow strength is highly sensitive to chemistry, with material from heat #3 showing higher strength levels both as cold worked and aged. The results of chemical analyses are shown in Table II--heat #3 is consistently lower in β stabilizer concentrations and higher in the α stabilizer, Al. To determine the effect of

TABLE I

Representative Tensile Data

Heat #2 ST 1/2 hr. at 850°C, cold drawn to 96% RA.

7977

Hours Aged at 425°C	UTS(ksi)	RA(%)		
0	195	58		
8	26 7	13		
16	272	12		
16	274	7		

Heat #3 ST 1/2 hr. at 850°C, cold drawn to 96% RA.

Hours aged at 425°C	UTS(ksi)	RA(%)
0 .	210	51
0	210	55
6	295	4
12	306	6

Heat #3 ST 1/2 hr. at 850°C, swaged to 96.2% RA,
resolution treated.

UTS(ksi)	RA(%)
135	65
140	

Heat #3 ST 1/2 hr. at 850°C, swaged to 96.2% RA.

7978

Hours Aged at 425°C	UTS(ksi)	RA(%)
0	224	60
2	308	19
6	318	
12	324	16
12	316	17
12	322	12
30	329	

alloy chemistry, a series of six ingots is desired, decreasing the alloying additions of Cr, V, and Mo in 5% steps from the nominal Ti: 10 Cr, 7 V, 3.5 Mo, 3 Al to Ti: 7.5 Cr, 5.25 V, 2.75 Mo, 3 Al.

TABLE II
Chemical Analysis

	Al	V	Cr	Мо	Fe	Cu	N	0	Н
Heat #2	2.73%	6.74%	9.80%	3.39%	0.07%	Nil	0.011%	0.116%	0.0136%
Heat #3	3.05	6.99	8.86	2.91	0.07	Nil	0.016	0.146	0.0163

Average fracture strengths, applying the Bridgman correction for triaxiality [7], have been calculated for the various classes of aged material and appear in Table III. The fracture strength of the swaged material is significantly higher than the others, indicating the importance of the processing mode. Furthermore, as seen in Figure 3b, the fracture strength of the aged material follows the modified Petch equation with the same slope as the unaged material. Thus the aging temperature is below the recovery temperature and the cellular barriers to dislocation motion appear unchanged. As seen in comparing Figures 3a and 3b, aging and the mode of deformation control the friction stress, σ_{y_0} or σ_{f_0} , in each case and have little effect on the dislocation pinning term κ'_{y_0} or κ'_{f_0} . In the aged material, σ_{f_0} is relatively constant for a given alloy composition and mechanical processing and independent of heat treatment. For the limiting case of no work hardening, the yield stress and the UTS are the same, and

$$\sigma_{f} = \frac{\sigma_{y}}{1 - RA} = \frac{\sigma_{y} + K'_{y}(1 - CW)^{-1/4}}{1 - RA}$$

where RA is the reduction of area. Thus a trade-off between UTS and RA exists, and combinations of strength and ductility can be manipulated within a wide range by varying the amount of cold work and the aging treatment.

TABLE III

Spe	ecimen C	lass		lverage Strength	(ksi)
96.2%	Swaged:	Heat	#3	356	
96%	Drawn:	Heat	#3	309	
95%	Drawn:	Heat	#2	294	
80%	Drawn:	Heat	#2	271	
63%	Drawn:	Heat	#2	265	

Effects of cold work on polygonized and α - β structures

Specimens were prepared by solution treating at 850°C, water quenching, cold rolling 65% at room temperature, polygonizing at 700°C for 20 minutes, and aging at 375°C. Contrary to a similar Soviet investigation [3,4], maximum hardness was less for this material, and it showed earlier overaging (Figure 7). Solutionizing at 750°C in the α - β field produced a recrystallized duplex structure with continuous α on the β grain boundaries and small equiaxed grains of isolated α within the β grains. After 50% cold reduction this material showed higher aged hardness and more rapid aging kinetics than the all- β material (Figure 8).

Effects of cold-reduction temperature

As previously indicated [5], there is no significant hardness variation following cold rolling to 80% RA at temperatures between -60°C and 100°C. Extensive testing confirms this (Figure 9). Furthermore, there appears to be no effect from varying the rolling reduction between 5% and 20% per pass. Aging

kinetics and peak hardness remain unaffected.

Multi-step aging

The effect of multi-step aging on hardness and tensile properties was investigated. It is expected that a primary low-temperature aging treatment enhances the rate of precipitate nucleation, yielding a fine, uniformly dispersed second phase of either ω as found in Beta-III [8] or, more likely, $\beta_1 + \beta_2$ as observed in Ti: 16 V, 2.5 Al [9]. Upon the intermediate β_2 sites it should then be possible to preferentially nucleate and grow α at a higher aging temperature.

Duplex aging at 375°C/425°C was investigated through hardness measurements as illustrated in Figure 10. A decrease in peak hardness is noted, and aging kinetics are accelerated. Similar results have been previously noted in aging at 300°C/375°C and 425°C/475°C [5].

A limited number of multi-step aging experiments on tensile specimens from the remaining small amount of material from heat #3 were also conducted. Results appear in Table IV. Only specimens #1 and #3 show combinations of strength and ductility equal to those obtained by conventional aging at 425°C. The combination of 261 ksi and 55% RA is a particularly outstanding result. The reasons for the otherwise poor response include retrogression at the higher temperatures—the primary precipitate is smaller than the critical size at the elevated temperature—and coarsening of the precipitate during the secondary aging treatments. An exhaustive study of multi-step aging should, however, produce some enhancement of properties and prove economically desirable in sharply reducing required aging times.

TABLE IV Multi-Step Aging

Heat	#3	swaged	96.2%	RA
11Ca c	11.0	Swagca	30.20	1/1/1

	473F	Heat #3	S swaged 96.	2% RA		
Specimen No.	hr. at: 245°C	6/17F 325°C	698F 370°C	797 F 425°C	UTS (ksi)	%RA
1	1	8		Marketon .	261	55
2	1	8	2	_	296	6
3	_	8	2	****	290	20
4	1	8		2	307	4
5		8		2	318	7
6	_	_	4	2	323	6

STRESS CORROSION

Stress corrosion tests were conducted in four-point bending of 0.14 inch thick strip specimens of TS6 and β -120VCA and in uniaxial tension of 0.050 diameter tensile specimens of TS6 in solutions of H₂O-3% NaCl and CH₃OH-1% HCl at 25°C. No failures were observed after 300 hour tests on TS6 cold worked 70% or 97%, aged up to eight hours at 425°C, and tested at 185,000 psi in the salt solution; nor on unaged specimens tested at 150,000 psi in the methanol-HCl solution. These conditions are thus presumably below the threshold for stress corrosion susceptibility. Such results for the salt solution are expected since TS6 has low aluminum content, contains an appreciable amount of the isomorphous beta stabilizers molybdenum and vanadium, and the specimens were not precracked.

Data from tests between 160,000 psi and 190,000 psi in methanol-HCl appear in Figure 11. Some scatter is observed in the data due to the statistical nature of the phenomenon; nevertheless, the significant trends can be identified. TS6 shows decreasing sensitivity to stress corrosion with increasing amounts of

cold work and increasing sensitivity as aging time increases. This is anticipated since susceptibility to stress corrosion is suppressed by homogeneity. The presence of a small amount of water also inhibits the process--TS6 swaged 96.2%, aged twelve hours at 425°C and tested in methanol-1%HC1-1%H₂O at 185 ksi failed after thirty minutes in contrast to eight minutes for similarly prepared specimens tested in methanol-1%HC1.

Specimens tested in tension fail somewhat earlier than similarly treated specimens tested in bending since the bend test is less severe—the tensile stress in the outer fiber being reduced after cracking begins, as opposed to being increased when tested in tension. In all cases, TS6 shows superior properties compared with similarly treated β -120VCA specimens.

CONCLUSIONS

One means of optimizing high strength and high ductility in metals can be characterized on the basis of five criteria: a metastable, single phase fcc or bcc starting material; a low strain-hardening exponent and good workability; a low stacking fault energy and high recovery temperature; a hardening mechanism active below the recovery temperature; and a low sensitivity of flow stress to temperature. By application of these criteria, thermomechanical processing of the \beta-Ti alloy TS6 has resulted in a material with an ultimate tensile strength in excess of 320,000 psi with 15% RA, exhibiting ductile behavior at elastic strains greater than 2%. Equivalent properties in other metals on a modulus basis would be 230,000 psi for aluminum alloys, 360,000 psi for copper alloys, and 680,000 psi for steels. While the present results are extraordinary, representing ductile behavior in metals at elastic strains in excess of 2.3%, they are not, we believe, unique to this specific alloy, for it is noted that a combination of 270 ksi UTS and 34% RA in a similarly treated Beta III has been

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recently reported [10]. These results are somewhat inferior to the properties we have obtained in TS6, but they support our contention that the extraordinary response to the thermomechanical treatments we have investigated is a microstructural effect not unique to a specific alloy. Rather they represent the behavior of a particular ultra-fine fibrous microstructure previously exploited only in heavily drawn pearlite.

The response of TS6 to a wide variety of thermomechanical treatments has been investigated. Combinations of strength and ductility can be manipulated within a wide range by varying the amount of cold work and the aging treatment. Polygonization prior to aging results in lower maximum hardness and more rapid kinetics. Extensive testing confirms the observation that the cold reduction temperature has no effect on resultant hardness between -60°C and 100°C. Multistep aging of TS6 produced lower peak hardness and accelerated aging response. Raising the UTS to 261 ksi while retaining 55% RA is extremely attractive and demonstrates the wide range of property manipulation available in this material as well as the need for continued research on the interaction and optimization of mechanical working and heat treatment. Mechanical properties are strongly dependent upon the mode of deformation during cold working--swaging producing an apparently larger amount of redundant work than drawing--and slight variations in alloy chemistry.

Stress corrosion tests in H_2^{0-3} NaCl and CH_3^{0H-1} HCl indicate the thresholds for susceptibility to be above 185 ksi and 150 ksi for the respective environments. In both cases TS6 shows superior performance compared with similarly treated β -120VCA.

A scale-up of ingot melting and processing techniques has been accomplished.

Ingots of TS6 weighing twenty-eight pounds have been prepared using a Cr-Mo

master alloy. A hot forging study has been conducted and a two-inch diameter;

rod has been extruded at 1800°F. This material has been successfully press-swaged to 20% RA and cold rolled to 90%; it shows slightly more rapid kinetics and overages at somewhat lower hardness levels than previous one-pound heats. In view of the difficulty of locating a satisfactory processor to accomplish the 95% cold reduction, separate industrial contracting to develop the capacity for heavy cold deformation of large section sizes is recommended if large-scale applications are to be pursued.

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BIBLIOGRAPHY

- 1. J. D. Embury, R. M. Fisher: Acta Met., 1966, vol. 14, p. 147.
- J. D. Embury, A. S. Keh, R. M. Fisher: Trans. TMS-AIME, 1966, vol. 236,
 p. 1252.
- R. M. Lerinman, K. I. Khvostyntsev, et al.: Fiz. Metl. Metalloved., 1966,
 vol. 22, p. 591.
- 4. R. M. Lerinman, G. V. Murzayeva: Fiz. Metal. Metalloved., 1968, vol. 25, p. 924.
- D. H. Avery, J. Greene, R. Wallace: Thermomechanical Processing β
 Titanium. Army Materials and Mechanics Research Center, AMMRC CTR 72-2,
 February 1972.
- 6. A. Ball, F. P. Bullen, F. Henderson, H. L. Wain: Phil. Mag. 1970, vol. 21, p. 701.
- 7. P. W. Bridgman: Trans. ASM, 1944, vol. 32, p. 553.
- 8. L. A. Rosales, A. W. Sommer, et al. in <u>Titanium Science and Technology</u>.

 New York: Plenum, 1973, vol. 3, p. 1813.
- 9. E. L. Harmon, A. L. Troiano: Trans. ASM, 1971, vol. 53, p. 43.
- 10. V. C. Peterson, F. H. Froes, R. F. Malone in <u>Titanium Science and</u>
 Technology. New York: Plenum, 1973, vol. 3, p. 1969.

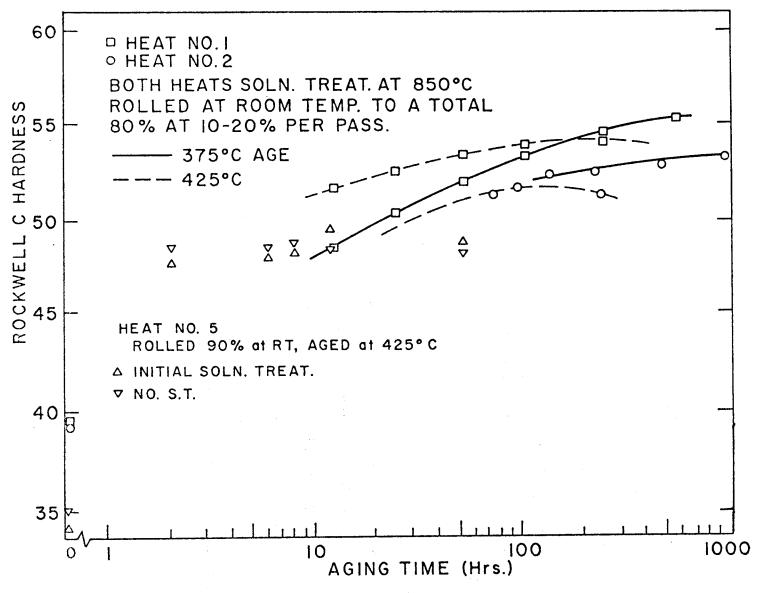


FIGURE 1. COMPARISON OF HARDNESS AS A FUNCTION OF AGING TREATMENT FOR HEATS #1, 2, AND 5.

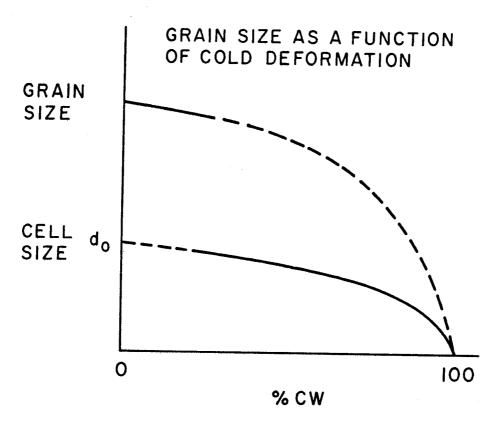


FIGURE 2. GRAIN SIZE AND SUBGRAIN SIZE AS A FUNCTION OF PERCENT COLD WORK. do IS THE HYPOTHETICAL SUBCELL SIZE AT 0% COLD WORK. THE SOLID LINES INDICATE THE REGIONS OVER WHICH EACH STRUCTURE IS DOMINANT.

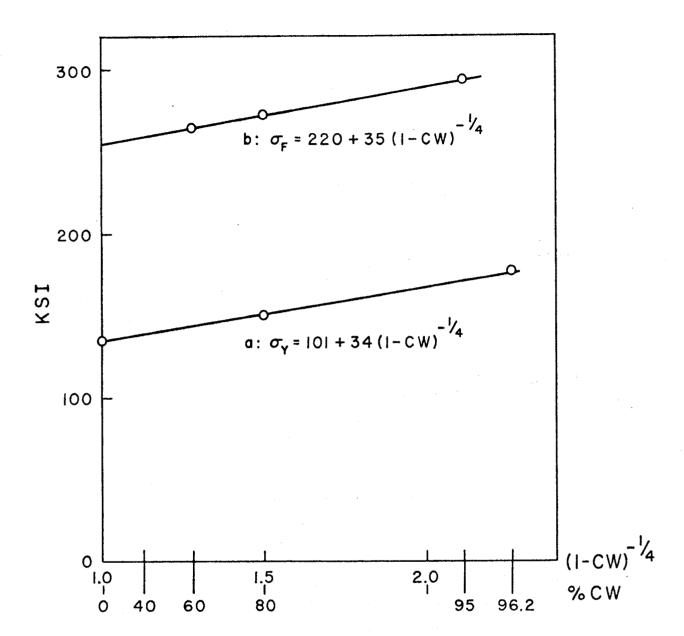


FIGURE 3. a: .2% YIELD STRESS OF UNAGED SWAGED MATERIAL FROM HEAT #3 AS A FUNCTION OF THE AMOUNT OF COLD WORK PRIOR TO TESTING.

b: AVERAGE FRACTURE STRENGTH OF DRAWN AND AGED MATERIAL FROM HEAT #2.

. TENSILE PROPERTIES OF SWAGED AND DRAWN TS 6

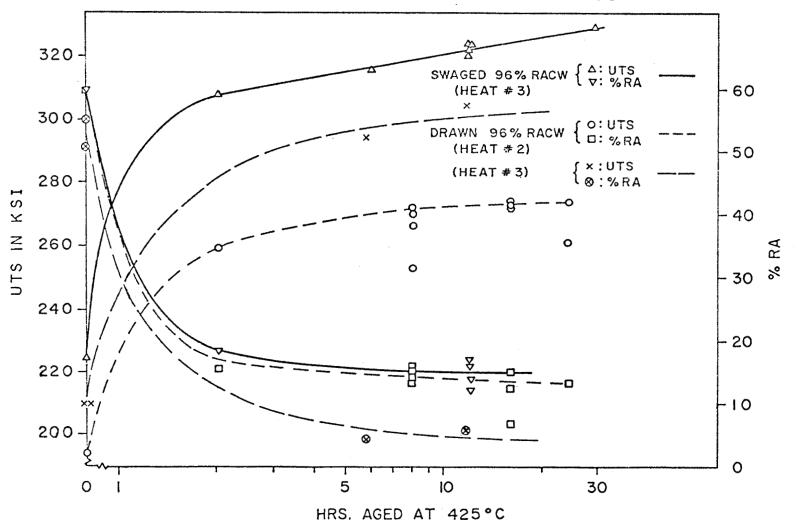


FIGURE 4. TENSILE PROPERTIES OF SWAGED AND DRAWN TS6.

TENSILE AXIS



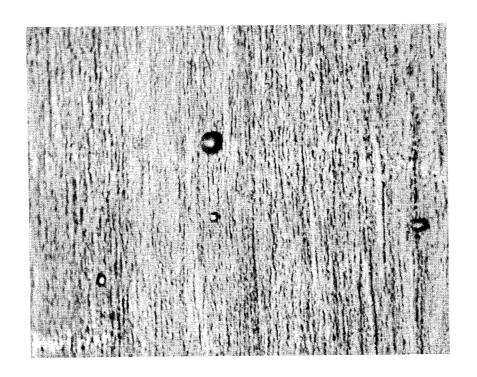


FIGURE 5. LONGITUDINAL SECTION OF NECKED SPECIMEN.

SWAGED 96.2% RACW

AGED 6 HR. @ 425°C Mag: 2000X

UTS = 328 ksi

PRIOR TO FRACTURE, SPHERICAL VOIDS ARE GENERAL THROUGHOUT CENTER SECTION.

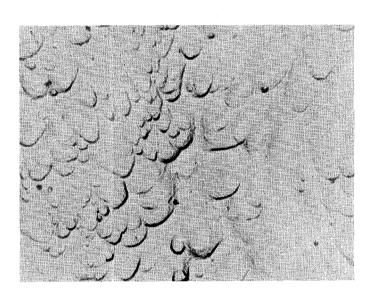


FIGURE 6. WOLF'S EAR FRACTURE SURFACE
SWAGED 96% RACW
AGED 12 HR. @ 425°C
UTS = 320 KSI
CARBON REPLICA: 7200X

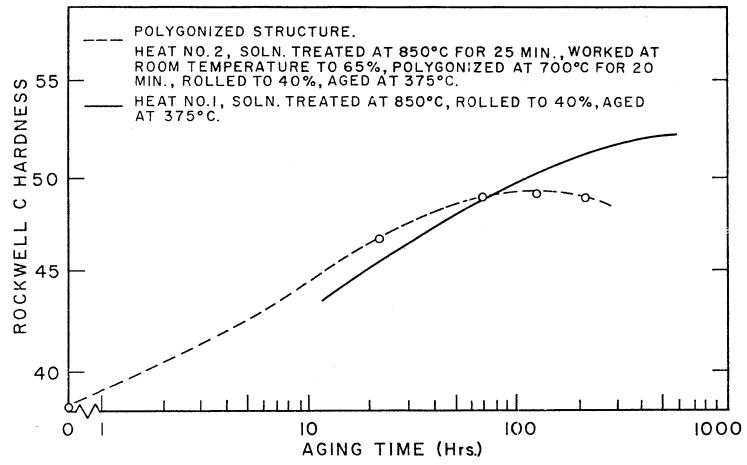


FIGURE 7. COMPARISON OF THE EFFECTS OF THERMOMECHANICAL PROCESSING ON POLYGONIZED AND SOLUTION TREATED TS6.

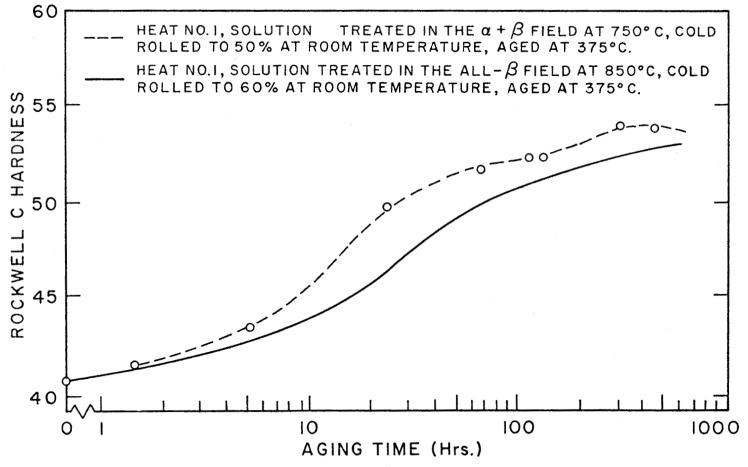


FIGURE 8. COMPARISON OF THE EFFECTS OF THERMOMECHANICAL PROCESSING ON a + B AND B TS6

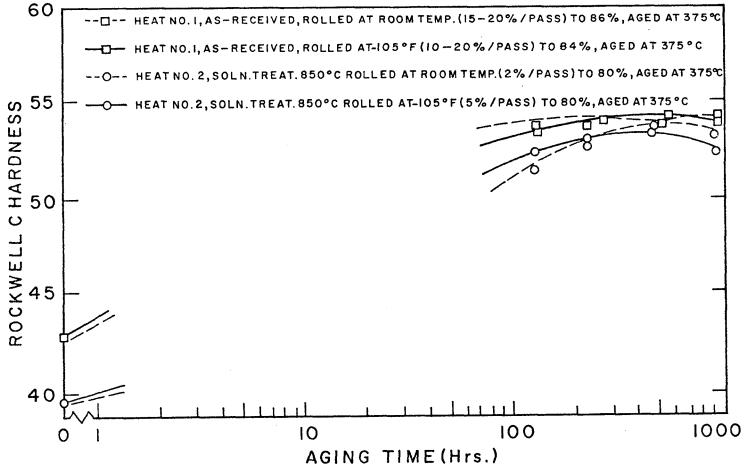


FIGURE 9. COMPARISON OF THE EFFECTS OF COLD REDUCTION TEMPERATURE ON THE RESPONSE TO THERMOMECHANICAL PROCESSING.

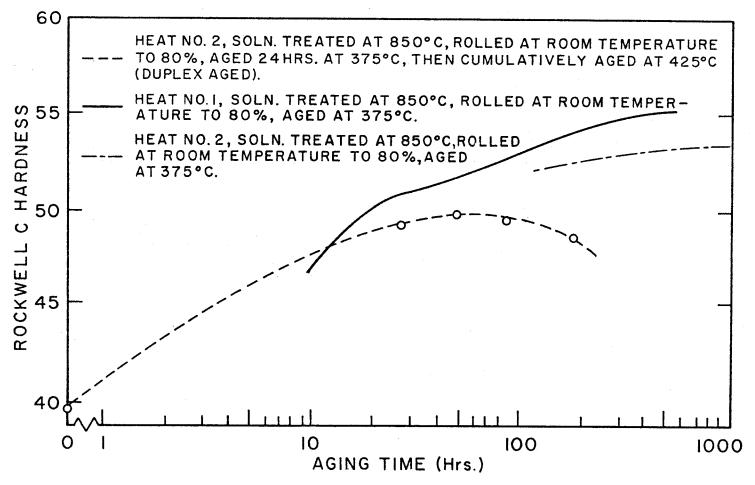


FIGURE 10. COMPARISION OF THE EFFECTS OF SINGLE AND TWO STEP AGING TREATMENTS.

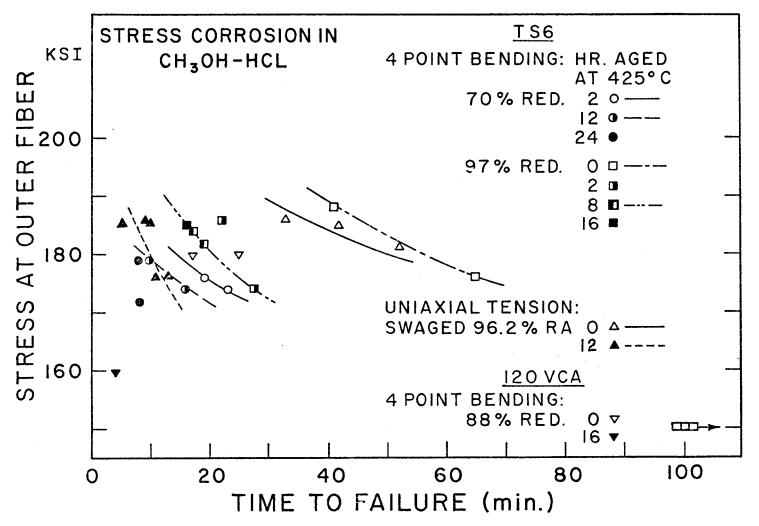


FIGURE 11. STRESS CORROSION PROPERTIES OF TS6 IN CH3OH-HCL.

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A set of criteria for the desi proposed. By application of these crite in the attainment in a β -Ti alloy of ten a cup-cone ductile shear failure of 55% in the second control of 55% in the	ria, thermome sile strength RA to 325,000	echanical property of the control of	rocessing has resulted from 261,000 psi with 15% RA.	
The response of the β alloy TS wide variety of thermomechanical treatme	6, (Ti: 10 c nts has been	r, 7 V, 3.5	5 Mo, 3 Al), to a	

effects of cold work on polygonized structures, the effects of cold reduction temperature, and multi-stage aging. A scale-up of ingot melting and processing techniques has been accomplished.

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